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#### DILUTE ACID PRETREATMENT RESEARCH

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#### ABSTRACT

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The dilute acid pretreatment of biomass (aspen wood and wheat straw) at higher solids concentrations (up to 40 wt%) can significantly improve the economics of this pretreatment. In the present study, the hemicellulosic component of the biomass was released as monomeric sugars (mainly xylose) in a concentrated liquid stream. Some loss of potentially recoverable xylose occurred due to conversion to furfural and entrapment of xylose in the residue. The cellulose remaining in the residue from both aspen and straw pretreated at either 140 or 160 °C was highly digestible with commercially available cellulase enzymes. A somewhat higher acid consumption for wheat straw compared with that for aspen can be alleviated by a cation removal step, and the relatively low yield of solubilized xylose (70-80%) should be improvable by further optimization of the pretreatment conditions.

#### INTRODUCTION

The prehydrolysis of selected biomass (wheat straw and aspen wood) with dilute sulfuric acid at temperatures higher than 120 °C is very effective for increasing the enzymatic digestibility of the cellulose component in pretreated residues by a cellulase enzyme complex produced by the fungus Trichoderma reesei (1-16). The dilute acid pretreatment process which was pioneered for high temperatures (i.e. 160 °C and above) by Grethlein and others (3-7,16) effectively breaks the bonds in the lignin-hemicellulose shield in agricultural residues and hardwoods. This pretreatment hydrolyzes glycosidic bonds in hemicellulose, as well as lignin-hemicellulose bonds and perhaps lignin bonds, which then leads to solubilization of hemicellulosic sugars and an increase in porosity of plant cell walls (7), thus increasing the accessibility of cellulose fibers to hydrolytic enzymes.

The operating costs of this pretreatment are very sensitive, however, to the consumption of steam, which is needed for heating the biomass and

dilute acid to elevated temperatures. The steam consumption can be lowered by the recycling of hot hydrolyzate and by incorporation of heat saving devices into the process, but the simplest solution is to increase the dry weight concentration of solids in the reactor (Fig. 1). Additional benefits stemming from this approach are the increase in concentration of solubilized hemicellulosic sugars and the potential decrease in the consumption of the acid catalyst. The mechanical properties of mixtures of biomass and dilute acids change dramatically as the concentration of solids is increased from approximately 10 wt%, where the mixtures behave as slurries and are easily stirred with conventional impeller mixers, to concentrations exceeding 15 wt%, at which concentrations the substrates are essentially wet solids. In view of this, the kinetics of the dilute acid pretreatment of wheat straw and aspen wood meals with dilute sulfuric

acid was investigated at solids concentrations between 20 and 40 wt%

using unstirred pipe reactors at 140 and 160 °C and compared to results obtained previously (1,2) with stirred slurries at 10% concentration of solids.

#### **EXPERIMENTAL**

## Substrates and other materials

Baled wheat straw was purchased at a local feed store. It was harvested locally in 1985 and visually inspected for absence of microbial decomposition. Debarked aspen logs 4 to 12 inches in diameter were obtained from a local lumber mill. A cellulase preparation (Celluclast 1.5L) produced by Trichoderma reesei was a kind gift of NOVO Industries, Ltd. The cellulase preparation was in liquid form, stabilized by the addition of glycerol. The specific activity on Whatman #1 filter paper averaged 71 international filter paper units (IFPU)/ml (17). glucosidase activity, measured using  $\rho$ -nitrophenyl- $\beta$ -D-glucopyranoside as a substrate (18) was approximately 21 IU per ml of the preparation carboxymethylcellulase activity measured with 7LF carboxymethylcellulose (Hercules Inc.) was approximately 932 IU per ml of the preparation (17). The enzyme was supplemented with fungal  $\beta\!=\!$  glucosidase (Novozyme 188, NOVO Ltd., specific activity 250 IU/ml) per manufacturer's recommendations. Both enzyme preparations were stored at 4 °C. The enzyme solutions retained their activities for several months. The remaining chemicals were purchased from national laboratory supply houses (Sigma Chemical Co., Aldrich Chemical Co. and VWR Inc.)

## Size reduction

Dry wheat straw was first shredded in a hammer-type mobile compost shredder (Mighty Mac, Amerind & MacKissic) equipped with a  $\frac{1}{2}$ " rejection screen, and subsequently milled in a laboratory knife mill (Wiley, model 4) equipped with 2 mm rejection screen. The milled straw was used directly in chemical pretreatment studies.

Air dried, debarked aspen logs were coarsely chipped using a Brush Bandit mobile knife chipper (Foremost, Inc.) and chips were then milled in the above mentioned laboratory knife mill or a rotary knife mill

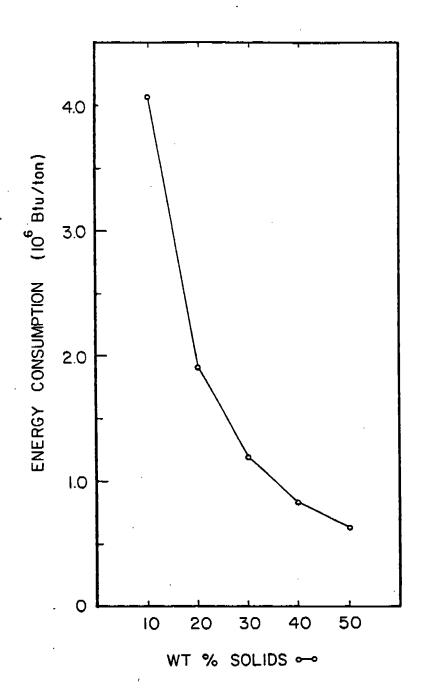


Figure 1. Approximate energy consumption for heating wood and water from  $20\,^{\circ}\text{C}$  to  $140\,^{\circ}\text{C}$  as a function of dry solids concentration.

(Mitts and Merrill, model 10/12) equipped with 1/8 or 1/16" rejection screens. Milled aspen particles were separated by screening with a 20" Combs gyratory screening machine (Great Western Manufacturing Co., model V). The pretreatment experiments were performed with the 60-80 U.S. mesh fraction.

## Chemical pretreatments

All pretreatment experiments at high concentrations of solids (20-40 wt%) were performed in small pipe reactors (  $\frac{1}{2}$  " I.D. x 4" long) constructed from Carpenter 20 Cb-3 stainless steel. The reactors were clamped into a custom fabricated, horizontally split aluminum heating block which was heated from the top and bottom by electric strip heaters (Watlow Electric). The temperature was controlled by an electronic temperature controller (Yellow Springs Instruments, model 63 RC) to within ±1°C. Larger batches (approx. 100 g) of dry biomass were wetted with dilute sulfuric acid by blending them with premeasured amounts of acid in a 2 gallon stainless steel reactor (Carpenter 20 Cb-3. Parr Co.) equipped with a custom ribbon blender. The blending was performed at room temperature for approximately one hour to ensure uniform wetting of biomass with dilute acid. The acidified biomass was divided into pipe reactors which were then closed and placed into the preheated aluminum block. Independent experiments using a thermocouple inside one of the pipe reactors indicated that the contents reached the preset temperature in 3-5 minutes. All pretreatments were timed from the point at which the reactors reached the desired temperature. The reactors were left in the heating block for the required length of time, removed and immediately plunged into a bucket of cold water to quench the reaction. The contents of the reactors were then quantitatively emptied into a fritted Buchner funnel and washed with hot (85°C) water until the pH of the filtrate fractions reached 5.0.

The solid residue was stored frozen at -20 °C for digestibility studies or air dried at 45 °C for chemical analyses. Volumes of the combined washes were measured. One part of the combined washes was neutralized as soon as possible with calcium carbonate, filtered and the oligosaccharide components determined immediately. Another part of the combined washes was used immediately for determination of furfural and acetic acid. The final aliquot was acidified to 3-4% sulfuric acid (v/v), and monosaccharide components were determined by posthydrolysis at 121°C followed by neutralization with  $CaCO_3$  and analysis by ion moderated partition chromatography (1,2).

## Determination of the pH in acidified biomass

The pH of the entrained liquid phase was determined by placing the sample in the cylinder of a modified piston press (Specimen Mount Press, Buchler Ltd.). The piston was lowered into the cylinder and liquid was expressed through the perforated bottom. It should be noted that high  $(1,000-3,000\ psi)$  pressures were required for expression of appreciable amounts of liquids from the wet biomass. The pH of the liquid expressate was measured by a combination glass electrode coupled to a digital pH meter (Beckman/Altex, Model 4500). The electrode and the meter were calibrated with pH 1.3, 2.0, 4.0 and 7.0 buffers. It was

assumed that the pH of the acidified biomass was the same as the pH of a liquid expressate.

## Analysis of solid residues

Dry weights (by air drying at 105 °C to constant weight) and Klason lignin were determined by standard methods (19,20). Ash and silica content were determined by gravimetric analysis according to A.O.A.C. methods (21). Cation content of the ash was determined by dissolving 100 mg of ash in 100 ml of 2% HNO $_3$  for 2 hours at 80 °C followed by analysis of the solubilized cations with an International Laboratories Plasma 100 ICP emission spectrophotometer. Acid insoluble ash was determined gravimetrically.

Anhydrosugars in the solids were determined by a procedure slightly modified from that developed at the U.S. Forest Products Laboratory (19) as described previously (1).

## Chemical analyses of liquid residues

Furfural and acetic acid in acidic combined washes were determined by gas chromatography on a Carbopack C/ 0.3% CW 20M/0.1%  $H_3PO_4$  packed column (6 foot x 2mm, Supelco) as described earlier (1,2).

Analysis of oligosaccharides was by HPSEC on Toyo-Soda TSK 1000 PW and G2000 PW columns. Subsequently, the oligomers were converted to monomers, and the total monomer composition determined by ion moderated partition chromatography using Bio-Rad HPX-87P columns as described previously (1,2).

## Determination of enzymatic digestibility.

Biomass residues from dilute acid prehydrolysis were tested for in vitro digestibility using the NOVO Celluclast 1.5 L cellulase preparation supplemented with  $\beta$ -glucosidase (Novozyme 188). The enzyme hydrolysis products were assayed for glucose and other sugars using a glucose analyzer (Yellow Springs Instruments Inc., model 27) and ion moderated partition chromatography on Bio-Rad HPX-87P columns (1,2).

Enzymatic digestions were performed at concentrations of cellulose equal to 10 mg/ml in 50 mM sodium citrate buffer, pH 4.8. The mixtures were incubated at 50 °C with gentle agitation. Assay mixtures contained tetracycline (40  $\mu g/ml$ ) to minimize bacterial contaminations. Approximate cellulase loadings of 33 IFPU/g of cellulose and a  $\beta$ -glucosidase supplement of 33 IU/g of cellulose were used in all experiments. This enzyme loading was found necessary for complete digestion (95%) of an external standard ( $\alpha$ -cellulose, from Sigma Chem. Co.) in 72 hours. The biomass samples were incubated for a period of 72 to 96 hours until release of soluble sugars from the digested samples ceased.

## Removal of cations

Removal of cations from wheat straw was performed with buffered

ethylenediaminetetraacetic acid (EDTA) solutions. 150 g of ground dry wheat straw was placed in a 4 liter suction flask with a magnetic stirrer and suspended in 3 liters of 0.5%  $Na_{2-3}$  EDTA solution, pH 7.0. The flask was stoppered and the slurry was evacuated for approximately 5 min to remove part of the air entrapped in the wheat straw. When the vacuum was applied, numerous air bubbles formed on the surface of the wheat straw and the straw floated to the top of the liquid where it formed an unstirred mat. The vacuum was therefore released and the straw that sank back into the liquid was stirred for several minutes under atmospheric pressure. The slurry was subjected to several cycles of exposure to vacuum and atmospheric pressure until the straw ceased floating to the top of the liquid while under vacuum. The vacuum was released and the slurry was stirred for 24 hours at room temperature. The slurry was filtered through muslin cloth held in a Buchner funnel. Filtrate was discarded and the wet straw was resuspended in 3 liters of 0.5% Na<sub>2-3</sub> EDTA solution. The straw was then re-extracted in the same manner as described above, once with 0.5% EDTA solution for 24 hours, once with 0.1% EDTA solution, and three times with deionized water. The extracted straw, with the cations removed, was finally air dried for several days at 45 °C until it reached constant weight.

#### RESULTS AND DISCUSSION

In this study, the effects of concentration of solids on the kinetics of dilute acid pretreatment of selected biomass substrates (wheat straw and aspen wood) were investigated. Both substrates were reduced to small particles prior to chemical pretreatment studies in order to minimize heat and mass transfer problems connected with the use of large biomass pieces (chips). The particle size distribution of the biomass was held constant between this study and our previous investigations which were conducted at a lower solids concentration of 10 wt%, in conventional stirred reactors (1). The change in the consistency of dilute acid biomass mixtures from slurries to wet solids resulting from increasing the concentration of biomass above 10 wt% necessitated a change in the The conventional impeller mixer cannot be used for reactor design. effective mixing of wet biomass particles and has to be replaced by specialized blenders for mixing of wet solids. We used small unstirred pipe reactors heated by an aluminum block for the present study, primarily because they could be heated and cooled rapidly and several inexpensive reactors could be operated simultaneously. The biomass and dilute sulfuric acid were thoroughly blended before pretreatment by mixing the two components at room temperature in a two gallon stainless steel reactor (Parr Co.) equipped with a ribbon blender. Each batch of blended material was then divided into the pipe reactors and used for a series of pretreatment cooks. Variation in hydrogen ion concentrations were thus minimized within a series and temperature effects for each concentration of solids and sulfuric acid could be studied. The two temperatures (140 and 160 °C) used in the present study were chosen on basis of previous results (1,2) which have shown that the hemicelluloses (xylan) are hydrolyzed rapidly and completely at these temperatures. Since there is a strong correlation between the extent of xylan removal and enzymatic digestibility of the cellulose in the pretreated biomass solids (2) (Fig. 4), the cellulose becomes highly

digestible after relatively short pretreatment times at these temperatures (2), (Figs. 2-4).

The original aims of the current study were to determine the acid loading required to match the pretreatment times (i.e. approximately 30 min. at 140 °C and 10 min at 160 °C) obtained previously with dilute (10%) slurries at pH 1.5 and also to study the effects of increased concentrations of solids on the rate of xylan removal and enzymatic digestibility of cellulose at a pH (1.5) which was near that used in the previous studies (1,2). These objectives were only partially met, mainly due to the difficulty of obtaining accurate measurements of the pH of wet biomass solids. The problem was finally solved by expressing the pretreatment liquor from the biomass at high pressures (1,000 - 3,000 psi) and determining the pH of the liquid phase with a calibrated pH electrode, but in the process many pretreatment runs were completed which matched the reaction times of the pretreatments at 10% solids but not the pH of those experiments. Also the results for each substrate began to diverge from previous results as is discussed below.

## Aspen wood

Chemical analysis was performed on aspen wood particles prior to the chemical pretreatment. The results are summarized in Table 1. The results agree well with our previous data (2). The cation content of ash was also determined and is shown in Table II.

The results of kinetic experiments performed at 140 and 160 °C are shown in figure 2. Previous results, obtained in a stirred reactor with 10 wt% solids, are shown as curves numbered 1 for comparison. correlation between the removal of xylan and enzymatic digestibility of cellulose in pretreated aspen wood is shown in Figure 4. The results clearly show that the dilute acid pretreatment can be readily extended from 10 to 40 wt% concentration of solids at both temperatures. There is a slight drop in xylan removal and enzymatic digestiblity of cellulose for 40 wt% solids pretreated at 140 °C (curve 5, Fig 2) but after a slightly longer reaction time (60 min) cellulose in these residues becomes as digestible as in other experiments. The 40 wt% solids pretreated at 160 °C do not deviate from other runs, therefore incomplete wetting of the solids can be ruled out as a reason for the drop in the rate of xylan removal in the 40 wt% solids pretreated at 140 °C. Perhaps the decrease in catalytic activity of hydronium ions caused by minor components, such as ash, starts to manifest itself as will be discussed below. Unfortunately, due to difficulties in pH measurements practically all experiments at higher concentrations of solids were performed at the lower pH (1.1-1.3) than the 10 wt% solids control (pH=1.45) and limited conclusions can be made concerning effects of increased concentrations of solids on the kinetics of pretreatment at the constant pH of the liquid phase.

A single set of kinetic experiments was performed at the end of a series with 30% solids and the same acid concentration (0.45%) as was used previously (2) with 10% slurries. The results (Fig 2, curve 6) indicate that the consumption of sulfuric acid catalyst by aspen wood is extremely low. The pH of the liquid phase was the same as for 10% wood

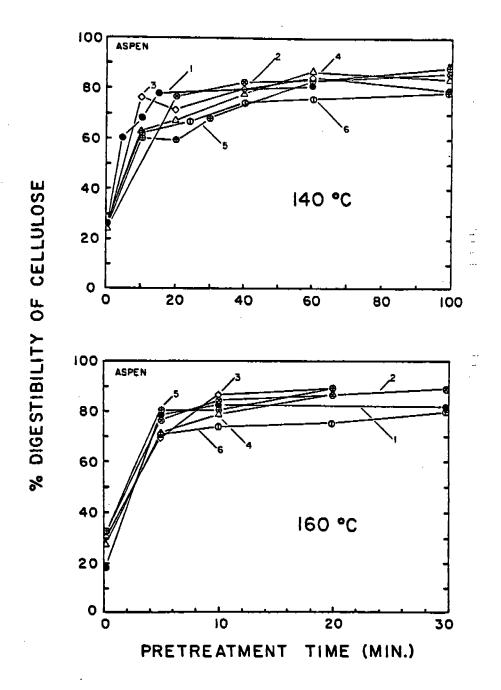


Figure 2. Enzymatic digestibility of chemically treated aspen wood meal using NOVO Celluclast 1.5L supplemented with beta-glucosidase as a function of time of pretreatment at  $140^{\circ}\text{C}$  and  $160^{\circ}\text{C}$ . Curve 1 represents  $10^{\circ}\text{K}$  solids treated with  $0.45^{\circ}\text{K}$  (v/v) acid, starting pH 1.45; curve 2 represents  $20^{\circ}\text{K}$  solids treated with  $0.8^{\circ}\text{K}$  (v/v) acid, starting pH 1.2; curve 3 represents  $20^{\circ}\text{K}$  solids treated with  $0.6^{\circ}\text{K}$  (v/v) acid, starting pH 1.3; curve 4 represents  $30^{\circ}\text{K}$  solids treated with  $0.8^{\circ}\text{K}$  (v/v) acid, starting pH 1.1; curve 6 represents  $30^{\circ}\text{K}$  solids treated with  $0.85^{\circ}\text{K}$  (v/v) acid, starting pH 1.1; curve 6 represents  $30^{\circ}\text{K}$  solids treated with  $0.45^{\circ}\text{K}$  (v/v) acid, starting pH 1.4. The final pH of each of the cooks represented above was approximately 0.1 units higher.

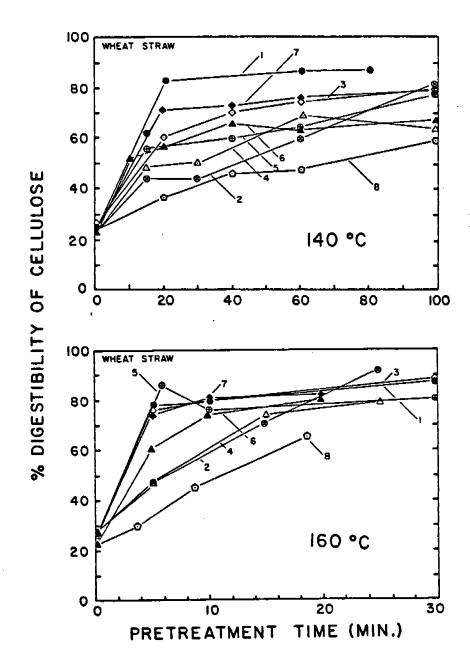


Figure 3. Enzymatic digestibility of chemically treated wheat straw using NOVO Celluclast 1.5L supplemented with beta-glucosidase as a function of time of pretreatment at  $140^{\circ}\text{C}$  and  $160^{\circ}\text{C}$  Curve 1 represents 10% solids treated with 0.5% (v/v) acid, starting pH 1.5; curve 2 represents 20% solids treated with 0.7% (v/v) acid, starting pH 1.5; curve 3 represents 20% solids treated with 0.9% (v/v) acid, starting pH 1.3; curve 4 represents 30% solids treated with 1.2% (v/v) acid, starting pH 1.4; curve 5 represents 30% solids treated with 1.5% (v/v) acid, starting pH 1.2; curve 6 represents 40% solids treated with 2.0% (v/v) acid, starting pH 1.2; curve 7 represents 40% solids of EDTA-treated and extensively washed wheat straw treated with 0.85% (v/v) acid, starting pH 1.3; curve 8 represents 50% solids treated with 2.5% (v/v) acid, starting pH 1.4. The final pH of each of the cooks represented above was approximately 0.1 units higher except for the EDTA-treated straw which remained constant.

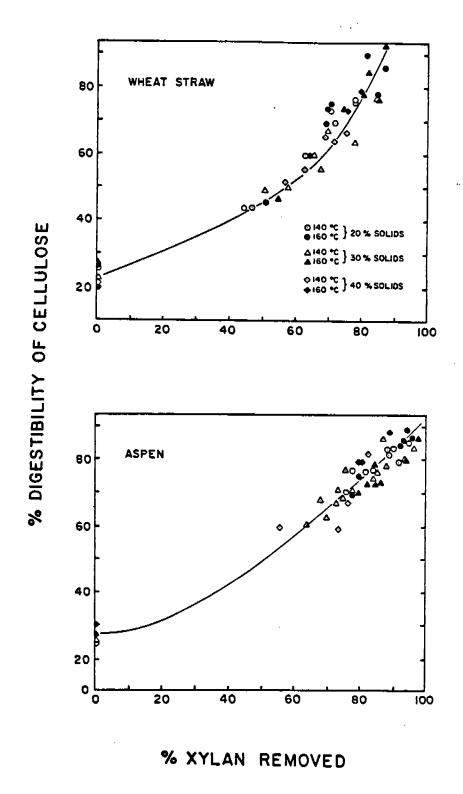


Figure 4. Enzymatic digestibility of chemically treated wheat straw and aspen wood using NOVO Celluclast 1.5L supplemented with beta-glucosidase as a function of acid catalyzed anhydroxylose removal.

slurries and the rate of xylan removal was also the same.

Therefore, the results clearly indicate that in addition to large savings in steam consumption (Fig 1), large savings (50% or more) in the consumption of acid catalyst are achievable by extending the pretreatment from 10% solids to 40% solids. The 10% solids needed 0.45% (v/v) sulfuric acid concentrations for approximately the same rate of xylan removal as 40% solids treated with 0.85% (v/v) sulfuric acid. Since the acid loading used in the current experiments was probably too high, further savings in acid consumption may still be achievable. The reason for low consumption of acid at higher concentrations of aspen wood is the low content of inorganic cations (ash) in the wood. The addition of the wood to dilute sulfuric acid therefore does not decrease the concentration and consequently catalytic activity of the hydronium ions to an extent the addition of wheat straw does, as is discussed below.

#### Wheat straw

Chemical analyses were also performed on wheat straw particles prior to chemical pretreatment (Tables I and II). Since a new batch of straw was used in this series of experiments, the results are slightly different from those reported previously (1,2). One striking difference between chemical composition of wheat straw and aspen wood is the much higher content of ash in wheat straw. Even though a large part of it is relatively inert silica (Tables I and II), the total quantity of cations is much higher in wheat straw than in aspen wood (Table I and II). While we have not completed the analysis of inorganic anions at the present time, results obtained by other investigators (22,23) indicate that there is an imbalance between inorganic cations and inorganic anions in biomass ash; part of the cations in biomass is therefore probably bound to carboxylic acid groups or is present as carbonates. The cations of such salts of weak acids are free to react with anions of stronger sulfuric acid and will partially neutralize it, thereby exerting a negative effect on the catalytic activity of the hydronium ions. This observation was made for dilute acid hydrolysis of red oak wood by workers at the U.S. Forest Products Laboratory (22). results (Fig. 3) confirm their observations. There is a signi There is a significant decrease in the rate and extent of xylan removal and in enzymatic digestibility of cellulose (see Fig. 4 and curves 2,4, and 8 in Fig. 3) as the concentration of solids is increased above 10%. The decrease in catalytic activity can be relieved by prior extraction of soluble ash from the straw with cold dilute EDTA solution and water at neutral pH (curve 7 in Fig. 3) or else the concentration of the catalyst has to be increased (Fig 3, curves 3, 5, and 6).

The EDTA extraction procedure is so mild that it should not produce any profound changes in polymeric components of cell walls. The removal of cations (see Table II) not only leads to a large drop in acid consumption as 2% (v/v) sulfuric acid was needed to bring the pH of 40 wt% wheat straw solids to pH 1.2 and 0.85% (v/v) sulfuric acid is needed to lower the pH of 40 wt% EDTA washed wheat straw solids to pH 1.3, but the xylan removal and enzymatic digestibility of cellulose improved significantly (compare curves 6 and 7 in Fig. 3) as well. Therefore our

Table I

CHEMICAL COMPOSITIO	N OF WHEAT STRAW (45°C air dried)	CHEMICAL COMPOSITION	N OF ASPEN WOOD {45°C air dried
18%	Klason lignin	18%	Klason lignin
7.2%	ash	0.43%	ash
2.5%	water	4%	water
41%	anhydroglucose	50%	anhydroglucose
19%	anhydroxylose	18%	anhydroxylose
3.5%	anhydroarabinose	4.0%	anhy droarabinose
2.2%	anhydrogalactose	2.0%	anhydromannose
		1.5%	anhydrogalactose
93.4% + Extractives		97.9% + E	xtractives

Table II

COMPOSITION OF ASH FROM WHEAT STRAW, EDTA WASHED WHEAT STRAW, AND ASPEN WOOD

(Weight Percent)

	,		
Component	Aspen	Wheat Straw	EDTA Washed Wheat Straw
Acid Insoluble Ash	N.D.1	58.9	97.0
Silicon Dioxide *	4.7	37.5	97.0
Mg	4.9	0.7	0.09
Ca	30.0	3.9	0.38
K	14.0	31.0	0.
Na	0.3	2.2	0.1
Мо	0	O	0
\$ <b>r</b>	0.04	0	• 0
Zn	0.2	0.02	0
Ba	0.3	0.03	0
Co	0	0	0
Ni	0	0	0
Mn	0.1	0.02	0
Fe	0.2	0.04	0
Сш	0.06	0	0
Lf	0	0	0
	54.8%	96.8%	97.6%

<sup>1</sup>Not Determined
\*Silicon dioxide is part of acid insoluble ash.

results indicate that the removal of cations from wheat straw, perhaps by washing with water or spent acid, prior to the pretreatment, would be beneficial. It would not only decrease the rather high consumption of acid by the cations in wheat straw, which can be estimated as 4-5 wt% of dry straw, but it would also improve the rate of xylan removal and increase the enzymatic digestibility of the residue. However these gains will be at least partially offset by additional processing required. There seems to also be a significant effect of temperature on the rate of xylan removal and the degree of enzymatic digestibility of cellulose in pretreated wheat straw which was not observed with aspen wood. The extent of xylan removal and hence enzymatic digestibility of cellulose (Figs 3 and 4) in wheat straw prehydrolyzed at 140 °C is consistently lower than results obtained at 160 °C with the exception of 10 wt% solids. Perhaps the decreased catalytic activity of sulfuric acid caused by higher cation concentration in straw is more detrimental at 140 °C than at 160 °C and that fraction of xylan bonds which is difficult to hydrolyze at 140 °C but not as difficult at 160 °C (2) is only partially hydrolyzed at higher concentrations of solids and the lower temperatures.

#### CONCLUSION

In summary, our results indicate that the dilute acid pretreatment of aspen wood and wheat straw can be readily extended to operation at higher concentration of solids than previously employed. Significant savings in steam consumption can thus be realized. In the case of aspen wood a large decrease in the acid consumption can be realized as well, even though the acid concentration may have to be slightly increased at higher concentrations of solids to compensate for the consumption of the acid by cations present in the wood. The pretreatment produces a liquid stream of relatively concentrated sugars (Table 4), mainly xylose. The analysis of the liquid streams by HPSEC and ion moderated partition chromatography (data not shown) indicate that the sugars are produced in the monomeric form and the concentration of oligomers does not increase as the concentration of solids is increased from 10 to 40 wt%. The sugar reversion reaction, therefore, does not seem to be significant at the concentrations of solids investigated so far.

The solid residues (Table 3) are mainly cellulose and lignin with small amounts of residual xylan. The pretreatment thus accomplishes at least partial separation of the three major components of biomass. Lignin can be easily separated by filtration after enzymatic hydrolysis of dilute acid pretreated cellulose or else separated from cellulose by additional chemical extractions. One problem which remains to be solved is a relatively low yield of solubilized xylose (70%-80%) which is partially converted to furfural and partially remains in the residue. However, the results by other investigators on the production of xylose by dilute acid prehydrolysis of biomass (22) indicate that improved xylose yields should be readily achievable by further optimization of pretreatment conditions.

Table III

PRETREATED SOLID RESIDUES (140 - 160° C)

	Wheat Straw	Aspen
Dry Weight Losses (wt%)	34-39	26-32
Klason Lignin Removal (wt%)	0-5	0-5
Composition of the Residues (wt1)		
Anhydroglucose	54-57	60-66
Anhydroxylose	4-5	0-3
Klason Lignin	31-33	26-29
H <sub>2</sub> O	3-5	3-5
Total	92-100	89-103

Table IV

COMPOSITION OF LIQUORS (140-160° C, 20 - 40% SOLIDS)

	Wheat Straw	Aspen Wood
Total sugars (wt%)	3.7-7.7%	3.6-7.3%
Acetic Acid (wt%)	0.7-1.4%	0.8-1.6%
Furfural (wt%)	0.28-0.92%	0.30-1.2%
Relative Sugar Composition(wt%)		
Xylose	68-71%	74-79%
Glucose	16-18%	12-16%
Arabinose + Mannose	10-12%	5-9%
Galactose	3-4%	. 2-4%
	97-105%	93-108%

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